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## (54) Gas feeding device and deposition film forming apparatus employing the same

Gasversorgungsvorrichtung und ihre Verwendung für eine Filmabscheidungsanlage

Dispositif d'alimentation en gaz et appareillage de disposition de films l'employant

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**Description**

The present invention provides a system comprising a viscous organometallic compound and a gas feeding device for feeding a starting gas comprising said compound for deposition film formation by a chemical vapour deposition method.

The method of chemical vapour deposition starting with an organometallic compound is widely employed to deposit metal thin films or III-V group semiconductor thin films. Trimethylgallium (TMG) and trimethylaluminum (TMA) are widely used as organometallic starting materials and are liquid at room temperature. The liquid organometallic compound can be converted into a vapour by a flow of a carrier gas such as argon which is introduced into the liquid organometallic compound through a tube. The vaporised compound is then transported to a reaction vessel in which there is a space where the film is deposited.

Figure 7 illustrates a conventional gas feeding device in which an organometallic compound is converted into a vapour component in a stream of carrier gas. A metallic container (1) stores the organometallic compound (2) in the liquid state. A carrier gas (6) such as argon is introduced into the container through a metallic pipe (41) and forms bubbles (3) in the liquid organometallic compound. As the carrier gas bubbles through the liquid (2) it becomes saturated with the vapour of the organometallic compound, and the saturated carrier gas is withdrawn through a pipe (42) which is above the level of the organometallic compound (2) and is fed to a reaction vessel (not shown). For example the inserted pipe (41) has a diameter of 6 mm (1/4") and at a carrier gas flow rate of 1 to 100 sccm, the organometallic compound such as a TMG and TMA can be transported out in the carrier gas through the pipe (42) in an amount corresponding to its saturated vapour pressure.

In order to improve productivity and in particular to provide deposition-film-forming apparatus which can be used to deposit a thin film on a plurality of substrates at the same time, a correspondingly increased amount of starting gas is required. If carrier gas is introduced at a large flow rate, e.g. as much as from 1 to 10 l/min, through a single metal pipe as shown in figure 7 bubbling will not produce sufficient saturation of the organometallic compound, so that an increase in the rate of flow of the carrier gas will not produce a corresponding increase in the quantity of organometallic compound transported. In particular, where the organometallic compound is viscous, the bubbles formed by the metal pipe (41) become relatively large in size which causes pulsation of the flow of discharged gas, or in an extreme case the bubbles may become joined together to form a tubular gas flow path direct to the gas space above the liquid surface (2). For example, using a metal pipe (41) having a 6 mm (1/4") diameter, as the carrier gas flow rate is increased above 100 cc/min, its pattern of flow can change from bubbles to tubular form, so that the amount of organometallic compound transported is not proportional to the carrier gas flow rate. One possibility to be considered for transporting organometallic compounds at a high flow rate is to use a plurality of containers for the organometallic compound. However, in order to obtain the required amount of organometallic compound in carrier gas flowing at a rate of from 1 to 10 l/min, 10 to 100 containers are required based on the assumption that an individual containers can accommodate a flow rate of 100 cc/min of the carrier gas. In such a case the disadvantages are encountered that the size of the apparatus and its cost are increased, and it becomes more complicated to maintain.

Japanese Laid Open Patent Application No Sho-62-33769 discloses a metal tube having a tip perforated with a number of holes. This may be effective in the case of an organometallic compound of low viscosity such as TMG, but is not always effective for a high viscosity organometallic compound.

Japanese Laid Open Application No Sho-62-207870 discloses installation into a container for an organometallic compound of an ultrasonic wave generator having a magnetostrictive oscillator. In an example, a metal pipe for introducing a carrier gas is inserted into a gas phase. However, the content of gas which can cause cavitation is generally low, and generation of mist as a result of ultrasound cannot be expected. Although the patent publication contains a description of introducing a metal pipe for the carrier gas into the organometallic compound, sufficient cavitation effect is not expected in viscous organometallic compounds because at high flow rates of carrier gas bubbles are not readily generated as mentioned above.

Furthermore, in the case of organometallic compounds having a low viscosity such as TMG, the above cited Japanese patent publication No Sho-62-207870 discloses a method which effectively transports TMG at a high flow rate. This method, however, is not so effective for organometallic compounds such as DMAH which are of high viscosity because no measure is taken to convert the carrier gas into minute bubbles as it passes through the organometallic compound.

Japanese Laid Open Application SHO-60-131973 discloses a method for vaporising a liquid organometallic compound by bubbling a gas through the liquid using a bubble-forming device having a gas ejection portion provided with a plurality of gas-ejecting holes at its tip. Even with this method, however, for rapid formation of deposition film which requires the organometallic compound to be vaporised at large amounts, instability of feed may arise because bubbles from the large amount of gas introduced combine one with another to form larger bubbles and splashing of the liquid organometallic compound is caused when these large bubbles burst out of the liquid, and accumulations of the liquid can form in the feed pipe.

For the above reasons, there has been a no satisfactory gas feeding device which enables large amounts of starting gas to be derived from an organometallic compound of high viscosity.

The present invention provides a system as set out in claims 1, 3 and 4 of the accompanying claims.

The invention also provides a system as aforesaid when used to produce a starting gas which contains dimethylaluminum hydride in a carrier gas which is hydrogen.

How the invention may be put into effect will now be described, by way of examples, with reference to the accompanying drawings. In the following description dimensions in inches may be converted into centimetres by the relation one inch = 2.5 centimetres, and pressures in Torr may be converted into Pascal using the relationship 1 Torr = 133 Pascals.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic diagram illustrating an example of the gas-feeding system of the present invention.

Fig. 2 is a timing chart for explaining a method for driving the device.

Fig. 3 is a schematic diagram illustrating another example of the gas-feeding system of the present invention.

Fig. 4 is a schematic diagram illustrating a still another example of the gas-feeding system of the present invention.

Figs. 5A to 5D are schematic diagrams for explaining the mechanism of preferable selective formation of the deposition film by using the gas-feeding system of the present invention.

Fig. 6 is a schematic diagram of a deposition-film-forming apparatus to which the gas-feeding system of the present invention is applicable.

Fig. 7 is a schematic diagram of a conventional gas-feeding apparatus.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Preferable gas-feeding systems of the present invention are as described below.

The gas-feeding system of the present invention comprises a container having a space for discharging the starting gas containing an organometallic compound by introduction of a carrier gas; a gas-introducing means connected to the container and having a plurality of gas-inlet for introducing the carrier gas or the starting gas into the container to generate the starting gas; and a gas generation-accelerating means for accelerating the generation of the starting gas.

The gas-feeding system of the present invention is capable of feeding a large quantity of a starting gas stably by employing a plurality of the gas-introducing openings and the gas generation-accelerating means. The gas-feeding system of the present invention is capable of transporting a large quantity of starting gas even with a viscous organometallic compound without causing problem of disadvantageous size increase of the apparatus.

The deposition-film-forming apparatus which can be used according to the present invention comprises the aforementioned gas-feeding system, a reaction chamber connected to the gas-feeding device to receive the starting gas fed therein, and a gas-exhausting means connected to the reaction chamber to exhaust the reaction chamber.

The deposition-film-forming apparatus comprising the gas-feeding system of the present invention is capable of feeding stably a large quantity of a starting gas into the reaction chamber, allowing formation of deposition film of high quality at a high deposition rate.

An example of the present invention is described in detail by referring to figures.

As described later, dimethylaluminum hydride (DMAH) gives a deposit of Al or an Al-Si alloy in high quality merely by thermal reaction on a surface of an electron-donating material by using hydrogen as a reaction gas. The vapor pressure of the DMAH is approximately 1 Torr at room temperature, so that it is readily transported in comparison with TIBA (vapor pressure: approximately 0.1 Torr at room temperature) which has been used as the starting material for Al in CVD.

In the case of deposition-film-forming apparatus according to the vacuum CVD method in mass production, the amount of the carrier gas used is required to be as large as 1 to 10 l/min. DMAH, which has a high viscosity, involves the problems that the transporting gas flow tends to become pulsated to result in hindrance of the continuous transportation, or the gas flow becomes tubular as mentioned before.

Accordingly, this example has the constitution below.

Fig. 1 illustrates an example of the constitution of the gas-feeding system of the present invention.

The metallic container 10 is made of SUS. The inside surface of the metallic container is preferably coated with SiO<sub>2</sub> or the like because an organometallic compound such as DMAH and TMA will decompose at a metal surface in the presence of hydrogen even at room temperature to form unnecessary alkane such as methane and the like. At the bottom of the container 10, a plurality of pipes are arranged in horizontal direction in approximately equal distances with the gas discharging openings directed upwards. A carrier gas 6 is introduced from an inlet pipe 4 to form bubbles 3. The vapor of the organometallic compound 2 is transported along with the carrier gas through an outlet pipe 5 to a

reaction vessel (see Fig. 6). A gas-switch 19 selects the gas introduction routes from pipes 11 - 17 inserted into the organometallic compound 2 to introduce the carrier gas fed from the inlet pipe 4 to pipes 11 - 17 in pulse with delays in timing. The gas-switch is constituted, for example, of valve bodies placed in connection portions of the respective pipes such as electromagnetic valves operated by electric current; an effector for the valves; and a control means for the effector. Although seven pipes are shown in Fig. 1, the number of the pipes may be more or less than seven. The timing for switching is adjusted depending on the length of each pipe (11 - 17) from the gas switch 19 to the outlets. In this example, the gas-switch 19 serves as a gas-generation-accelerating means.

Fig. 2 shows a timing pattern for introduction of the carrier gas to the pipes 11 - 17. In Fig. 2, the hatched portions show the time of flowing of the gas in the respective pipes. The flow rates in respective pipes are adjusted to be equal at any time. In correspondence with the dimension of the pipes, the time  $T_1$  and  $T_2$  is decided so that the bubbles formed by the carrier gas in the organometallic compound may not communicate the outlets with the gas layer. In the timing chart of Fig. 2, the carrier gas flows through 6 pipes at any time as the simplest example, where the one sixth of the carrier gas introduced through the inlet pipe 4 is distributed to each of the six pipes 11 - 17.

In the case where the pipes have respectively one opening, the quantity of the carrier gas introduced into the respective pipes 11 - 17 is preferably not more than 100 cc/min for DMAH. In a more preferable example, where plural small openings, not a single opening, are provided for each of the tip of the pipes 11 - 17, the carrier gas flow rate in each pipe may be larger than 100 cc/min. When small openings of  $n$  in number are provided in each pipe, then the carrier gas flow rate in each of the pipes is up to  $n \times 100$  cc/min. Specifically, for each of the pipes 11 - 17 of 3/8 inch or 1/4 inch in diameter, the number is more preferably not more than 10. If the number is larger, the formed bubbles may combine with each other in high probability. At any rate, a measure should be taken so that the bubbles ejected from the tip of the pipe may not join together to form a tubular gas phase communicating with the above gas layer.

In the device shown in Fig. 1, bubbles are formed sequentially from the respective pipes 11 - 17 nearly continuously as a whole without pulsation of the starting gas 7 discharged from outlet pipe 5.

By use of the gas-feeding system of the example, the size of bubbles from the respective openings can be made smaller by appropriate selection of the flow rate of the carrier gas introduced through the plural gas-introducing means and intermittent feed of the carrier gas to respective gas-introducing member with a recess time of  $T_2$ , whereby the organometallic compound can be vaporized sufficiently into the gas, and a large quantity of the starting gas can be fed.

Fig. 3 shows another example of the constitution of the gas-feeding system of the present invention.

The metallic container 10 is made of SUS. The inside surface of the metallic container is preferably coated with  $\text{SiO}_2$  or the like because an organometallic compound such as DMAH, and TMA will decompose at a metal surface in the presence of hydrogen even at room temperature to form unnecessary alkane such as methane and the like.

A plurality of a small openings 305 are provided for gas discharge on the metal pipe 4 inserted in a organometallic compound. A carrier gas is ejected through each of the small openings 305. The small openings 305 may be provided either in a uniform interval or in irregular intervals. The pipe 4 inserted in the organometallic compound, although illustrated in linear form in Fig. 3, may be in a circular form or in a serpentine form so as to distribute the positions of the openings 305 uniformly in the organometallic compound container 10.

In particular, if the organometallic compound 2 is DMAH having a high viscosity, the carrier gas flow rate through the respective small openings 5 is preferably not more than 100 sccm. A larger flow rate than 100 sccm may destroy the bubbles of the carrier gas ejected from the small openings 305, forming an open gaseous path from the small openings 305 to the surface of the organometallic compound in a tubular form, which may retard the cavitation effect generated by a supersonic oscillator employed in this example.

The ultrasonic oscillation 8 exhibits a cavitation effect to generate fine bubbles, forming a mist of the organometallic compound from the surface of the organometallic compound effectively. The ultrasonic oscillator may be provided in plurality irrespectively of the one ultrasonic oscillator 8 in Fig. 3. Moreover, the ultrasonic oscillator may be installed on the side face of the organometallic compound. In this example, the ultrasonic oscillator 8 serves as a gas-generation-accelerating means for forming the starting gas effectively.

In this example, small openings provided and a limited flow rate of carrier gas ejected from the each small opening 305 realizes the generation of fine bubbles in the organometallic compound and formation of mist on the liquid surface. However, the effective generation of fine bubbles cannot be achieved sufficiently only by providing the small openings 305. In particular, for a viscous organometallic compound like DMAH, the carrier gas is desired to be ejected from the respective small openings 305 continuously and at a flow rate lower than a certain level.

In this example, DMAH having a high viscosity can be transported by the carrier gas flowing out from the organometallic compound contained in the metallic container 10 containing an amount corresponding to the saturated vapor pressure of DMAH by limiting the flow rate of the carrier gas ejected from small openings 305 to approximately 100 sccm and employing a cavitation effect of ultrasonic oscillation, even when the total carrier gas introduced to the metallic container 10 is in an amount as large as 10 slm (standard liter per minute).

No method has hitherto been found for transporting effectively an organometallic compound by such a large amount of a carrier gas. In particular, in the vacuum CVD method, the number of wafers used in one deposition batch depends

on the efficiency of transportation of a large amount of viscous DMAH. The use of the gas-feeding system of the present invention in a deposition-film-forming apparatus shown later in Fig. 6 makes feasible the deposition of an Al or Al-Si thin film on 100 - 200 wafers of 4 inches in one deposition batch at a high deposition rate.

In the gas-feeding device shown in the present example, a mist of an organometallic compound is generated in a great number of fine bubbles or in a gas phase of the organometallic compound-storing means by selecting appropriately the carrier gas flow rate for each small opening 305 (e.g., approximately 100 sccm or less), and further by causing cavitation by an ultrasonic oscillator installed in the metallic container containing the organometallic compound, thereby the organometallic compound can be contained in the carrier gas ejected from the container in an amount corresponding to the saturated vapor pressure.

A still another example of constitution of the gas-feeding system of the present invention is illustrated in Fig. 4.

An organometallic compound is stored in a liquid state in a metallic container 31. A gas is introduced through nozzles 30. Atomizers 34 (piezoelectric atomizers) employing a piezoelectric element as an ejection-energy-generating means spray the organometallic compound drawn up from the metallic container 31 in a mist state into a metallic chamber 33. The piezoelectric atomizer 34 is preferably provided in plural numbers. Five atomizers are provided in the example in Fig. 4. The atomizers 34 are connected to a driving circuit (DR) for supplying a driving signal to be controlled by the driving circuit. In this example, the piezoelectric atomizers 34 serve as a gas-generation-accelerating means for generating the starting gas effectively.

The organometallic compound is ejected in a size of several  $\mu\text{m}$  or less in a mist state from the piezoelectric atomizers 34. Although the atomizers 34 are installed at the bottom of the metallic chamber 33 in the figure, they may be installed at the side face of the chamber. An atomizer other than piezoelectric type may naturally be used.

The inside wall of the metallic chamber 33 is preferably coated with an insulating material such as  $\text{SiO}_2$  or the like because an organometallic compound such as TMA and DMAH will decompose at a metal surface of SUS in the presence of hydrogen even at room temperature to form unnecessary alkane such as methane and the like.

In such a device, a carrier gas 6 such as hydrogen, argon, nitrogen, and the like is introduced through an inlet pipe 37. The carrier gas comes to contain the organometallic compound in an amount corresponding to the saturated vapor pressure while passing through the metallic chamber where the organometallic compound is sprayed in a mist state, and flows out from an outlet pipe 9 as a starting gas 7. The organometallic compound is in a mist state having a size of several  $\mu\text{m}$  or less, which is contained in the carrier gas effectively.

The organometallic compound which has attached to the inside face of the metallic chamber 33 is recovered through a recovery pipe 36 to the metallic container 31 containing the organometallic compound.

The use of a piezoelectric atomizer allows spraying in a mist state even if the organometallic compound has a high viscosity, so that the organometallic compound can be contained in a carrier gas effectively independently of the viscosity of the organometallic compound. Since the carrier gas does not pass through a liquid, no fluctuation of the flow rate is caused of the carrier gas 7 containing an organometallic compound. Thus pulsation of the flow can be completely prevented which will be caused in transportation of an organometallic compound of high viscosity. By the method of transporting an organometallic compound of this constitution, the transportation of DMAH can be effected in the state that the carrier gas contains DMAH in an amount corresponding to the saturated vapor pressure thereof, even if the total flow rate of the carrier gas is as much as 10 slm (standard liter per minute).

No method has hitherto been found for transporting effectively an organometallic compound by such a large amount of a carrier gas. In particular, in conventional vacuum CVD method employing DMAH, the number of wafers usable in one deposition batch depends on the efficiency of transportation of a large amount of viscous DMAH. The use of the gas-feeding system of the present invention in a deposition-film-forming apparatus shown later in Fig. 6 makes feasible the deposition of an Al or Al-Si thin film on 100 - 200 wafers of 4 inches in one deposition batch at a high deposition rate.

The gas-feeding device shown in the example transports an organometallic compound in a liquid state from a container storing the organometallic compound, and ejects the liquid organometallic compound in a mist through an ejection means like a piezoelectric nozzle into a space, thereby discharging an introduced gas which contains the organometallic compound in an amount corresponding to the saturated vapor pressure thereof.

The gas-feeding system described above is suitably used with a deposition-film-forming apparatus described below.

Fig. 6 illustrates a schematic sectional view of a deposition-film-forming apparatus.

An external reaction tube 50 made of quartz forms a substantially enclosed space for deposition film formation. In the interior, a plurality of substrates 57 for formation of a metal film mainly constituted of aluminum, silicon or the like as the deposition film are fixed by a substrate holder 56 at a predetermined position. An internal reaction tube 51 made of quartz separates the gas flow in the external reaction tube 50. A metallic flange 54 closes or opens an opening of the external reaction tube 50. The substrates 57 are placed in the substrate holder 56 arranged in the interior reaction tube 51. The substrate holder 56 is preferably made of quartz.

In this apparatus, the temperature of the substrates is controlled by a heater 59. The pressure in the reaction tube 50 is controlled by an exhausting system connected through a gas exhausting outlet 53.

The apparatus shown by Fig. 6 has a mixer 62 which is connected with a first gas system for a gas fed from a gas-feeding device, and a second gas system for H<sub>2</sub> or the like, and further a third gas system for Si<sub>2</sub>H<sub>6</sub> or the like, from which the resulting starting gas mixture is introduced through a starting-gas-introducing pipe 52 into the reaction tube 50. The starting gas reacts at the surface of the substrates 57 while passing through the inside of the interior reaction tube 51 as shown by arrow marks 58 in Fig. 6, resulting in deposition of Al or Al-Si on the substrate surface. The gas after the reaction passes through a space formed by the internal reaction tube 51 and the external reaction tube 50 and is exhausted from an exhausting outlet 53.

To take in or out the substrates, the metallic flange 54 is lowered together with the substrate holder 56 and the substrates 57 by means of an elevator (not shown in the figure) to move it to a predetermined position.

With such an apparatus under the aforementioned deposition-film-forming conditions, satisfactory Al or Al-Si film can be formed simultaneously on all wafers in the apparatus.

As described above, the gas-feeding system of the present invention is particularly suitable for apparatuses for forming deposition films by use of an organometallic compound having a high viscosity like DMAH in a large amount. Naturally, the kind of the organometallic compounds, the constitution of the deposition-film-forming apparatuses or the like matters are not limited to the above examples.

According to the present invention, excellent deposition films can be formed in a large quantity at a high throughput since present invention enables stable transportation of a large amount of starting gas without problems such as size increase of the apparatus even when an organometallic compound having a high viscosity is employed. Thus the production cost of the semiconductor device can be reduced remarkably.

Specifically, the gas-feeding device illustrated in Fig. 1 is suitably applied to an apparatus for the vacuum CVD method illustrated, for example, in Fig. 6 which is capable of holding a large number of wafers (substrates) at a time and depositing Al or Al-Si. Since the deposition of Al or Al-Si undergoes by a surface reaction at a heated electron-donating substrate surface, Al or Al-Si can be deposited from DMAH and H<sub>2</sub> (or further with an Si starting gas such as Si<sub>2</sub>H<sub>6</sub>) by a hot wall type of the vacuum CVD method in which the substrate only is heated.

In the film formation, Al or Al-Si deposits to a thickness of 100 - 200 Å only on an electron-donating substrate under conditions of the reaction tube pressure of from 10<sup>-3</sup> to 760 Torr, the substrate temperature of from 270 to 350 °C, DMAH partial pressure of from 1 × 10<sup>-5</sup> to 1.3 × 10<sup>-3</sup> time the reaction tube pressure, (or further Si<sub>2</sub>H<sub>6</sub> partial pressure of from 1 × 10<sup>-7</sup> to 1 × 10<sup>-4</sup> time the reaction tube pressure). For improvement of film thickness uniformity in wafers, the reaction tube pressure is preferably from 5 × 10<sup>-2</sup> to 5 Torr, and the DMAH partial pressure is preferably from 1.3 × 10<sup>-5</sup> to 1.3 × 10<sup>-4</sup> time the reaction tube pressure. The preferable substrate temperature is from 270 to 300 °C for suppressing surface migration and preparing satisfactory continuous film.

Further, the gas-feeding device illustrated in Fig. 3 is also suitably applied to an apparatus for the vacuum CVD method illustrated, for example, in Fig. 6 which is capable of holding a large number of wafers (substrates) at a time and depositing Al or Al-Si. Since the deposition of Al or Al-Si undergoes by a surface reaction at a heated electron-donating substrate surface, Al or Al-Si can be deposited from DMAH and H<sub>2</sub> (or further with an Si starting gas such as Si<sub>2</sub>H<sub>6</sub>) by a hot wall type of the vacuum CVD method in which the substrate only is heated.

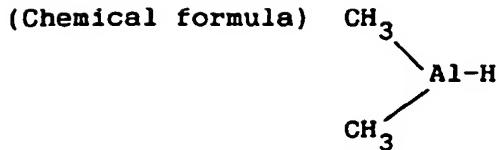
In the film formation, Al or Al-Si deposits to a thickness of 100 - 200 Å only on an electron-donating substrate under conditions of the reaction tube pressure of from 10<sup>-3</sup> to 760 Torr, the substrate temperature of from 270 to 350 °C, DMAH partial pressure of from 1 × 10<sup>-5</sup> to 1.3 × 10<sup>-3</sup> time the reaction tube pressure, (or further Si<sub>2</sub>H<sub>6</sub> partial pressure of from 1 × 10<sup>-7</sup> to 1 × 10<sup>-4</sup> time the reaction tube pressure). For improvement of film thickness uniformity in wafers, the reaction tube pressure is preferably from 5 × 10<sup>-2</sup> to 5 Torr, and the DMAH partial pressure is preferably from 1.3 × 10<sup>-5</sup> to 1.3 × 10<sup>-4</sup> time the reaction tube pressure. the preferable substrate temperature is from 270 to 300 °C for suppressing surface migration and preparing satisfactory continuous film.

Still further, the gas-feeding device illustrated in Fig. 4 is also suitably applied to an apparatus for the vacuum CVD method illustrated, for example, in Fig. 6 which is capable of holding a large number of wafers (substrates) at a time and depositing Al or Al-Si. Since the deposition of Al or Al-Si undergoes by a surface reaction at a heated electron-donating substrate surface, Al or Al-Si can be deposited from DMAH and H<sub>2</sub> (or further with an Si starting gas such as Si<sub>2</sub>H<sub>6</sub>) by a hot wall type of the vacuum CVD method in which the substrate only is heated.

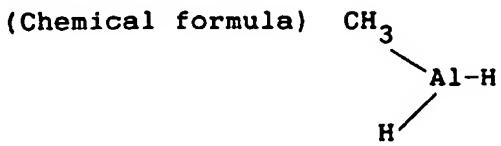
In the film formation, Al or Al-Si deposits to a thickness of 100 - 200 Å only on an electron-donating substrate under conditions of the reaction tube pressure of from 10<sup>-3</sup> to 760 Torr, the substrate temperature of from 270 to 350 °C, DMAH partial pressure of from 1 × 10<sup>-5</sup> to 1.3 × 10<sup>-3</sup> time the reaction tube pressure, (or further Si<sub>2</sub>H<sub>6</sub> partial pressure of from 1 × 10<sup>-7</sup> to 1 × 10<sup>-4</sup> time the reaction tube pressure). For improvement of film thickness uniformity, the reaction tube pressure is preferably from 5 × 10<sup>-2</sup> to 5 Torr, and the DMAH partial pressure is preferably from 1.3 × 10<sup>-5</sup> to 1.3 × 10<sup>-4</sup> time the reaction tube pressure. The preferable substrate temperature is from 270 to 300 °C for suppressing surface migration and preparing satisfactory continuous film.

The method for deposition film formation, namely, the CVD method utilizing an alkylaluminum hydride, particularly an alkylaluminum hydride having a methyl group is described in detail.

In this method, a gas containing an organometallic compound, specifically dimethylaluminum hydride (DMAH):



or monomethylaluminum hydride ( $\text{MMAH}_2$ )



- 15 is used as a starting gas containing at least one atom to be a constitutional element of the deposition film, or further a gas containing Si atom as a starting gas, or further additionally hydrogen as the starting gas are used. An Al or Al-Si film is formed selectively on the substrate by vapor deposition.

The substrate applicable to this method has a first substrate surface material for providing a surface for deposition of Al or Al-Si, and a second substrate surface material for non-deposition of Al or Al-Si. The first substrate surface material is composed of an electron-donating substance.

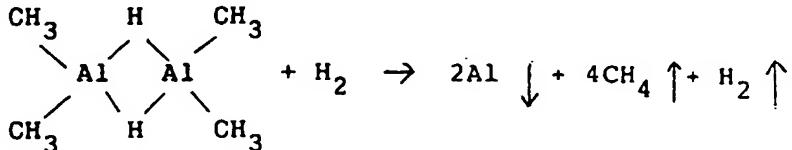
The electron-donating property is explained in detail.

An electron-donating material is such a material that free electrons exist or free electron is intentionally formed in the substrate: for example, a material having a surface on which a chemical reaction is accelerated by electron transfer with a starting gas molecule adhering on the substrate surface. Generally, metals and semiconductors are the examples. Metals and semiconductors having extremely thin oxide film on the surface are included thereto, since the electron transfer between the substrate and the adhered starting molecule occurs to cause the chemical reaction if the oxide film is extremely thin.

Specific examples thereof are semiconductors such as single crystalline silicon, polycrystalline silicon, amorphous silicon and the like; III - V group compound semiconductor and II - VI group compound semiconductor composed of binary, ternary or quaternary combinations of Ga, In, and Al as the Group III element and P, As, and N as the group V element; metals per se such as tungsten, molybdenum, tantalum, aluminum, titanium, copper, and the like; silicides of the above metals such as tungsten silicide, molybdenum silicide, tantalum silicide, aluminum silicide, titanium silicide, and the like; and metals containing any one of the above metal such as aluminum silicon, aluminum titanium, aluminum copper, aluminum tantalum, aluminum silicon copper, aluminum silicon titanium, aluminum palladium, titanium nitride, and the like.

On the other hand, the materials having a surface on which Al or Al-Si does not selectively deposit, namely non-electron-donating materials include usual insulating materials; silicon oxide formed by thermal oxidation, CVD, etc.; glass or oxide films such as BSG, PSG, BPSG, and the like; prepared by the thermal CVD, plasma CVD, vacuum CVD, ECR-CVD methods; and the like materials.

On the substrate of such constitution, Al or Al-Si deposits by only a simple thermal reaction in a reaction system of a starting gas and hydrogen. For example, the thermal reaction in the reaction system of DMAH and hydrogen is considered basically as below:



- 50 The DMAH has a dimer structure at a room temperature. The addition of  $\text{Si}_2\text{H}_6$  or the like causes formation of Al-Si alloy because the  $\text{Si}_2\text{H}_6$  having reached to the substrate surface decomposes by a surface chemical reaction and the resulting Si is incorporated into the film. From  $\text{MMAH}_2$  also, high quality Al could be deposited. However,  $\text{MMAH}_2$ , which has a vapor pressure as low as 0.01 to 0.1 Torr, cannot readily be transported in a large amount, thereby the upper limit of the deposition rate being several hundred Å/min or below. Therefore DMAH, which has a vapor pressure of 1 Torr at room temperature, can be preferably used.

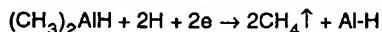
55

The aluminum film prepared by the above method assumes a single crystal structure, excellent in surface smoothness, anti-migration characteristics, etc., having low resistance and extremely superior characteristics for wiring and use of electrodes.

In deposition of an aluminum film from an alkylaluminum hydride, the temperature of the substrate is selected within the range of from the decomposition temperature of alkylaluminum hydride to be used to 450 °C, more preferably from 200 to 350 °C, still more preferably from 270°C to 350 °C.

The mechanism of aluminum deposition is considered as described below referring Fig. 5 at the moment.

When DMAH reaches, with its methyl group directed to the substrate sides, a substrate in a state that hydrogen atoms are attached on an electron-donating substrate, namely on a substrate having electrons (Fig. 5A), the electron on the substrate cuts a bond between aluminum and a methyl group (Figs. 5B and 5C). The reaction is as below.



10 The similar reaction proceeds with the hydrogen remaining on aluminum having deposited and having free electrons (Fig. 5D). If hydrogen atoms are deficient, a hydrogen molecule being the starting gas decomposes to supply hydrogen atoms. On the contrary, on a non-electron-donating surface, the above reaction does not proceed owing to lack of electrons, causing no deposition of aluminum. Incidentally, Figs. 5A to 5D are to aid understanding of the reaction mechanism, so that the numbers of H, e and Al are not consistent

15 Examples of experiment regarding gas transportation by using a gas-feeding device of the present invention are described below.

#### Experimental example 1

20 Into an organometallic compound contained in the metallic container 10 of Fig. 1, hydrogen gas was introduced through the metallic pipe 4 at a flow rate of L sccm. By utilizing the gas switch 19, hydrogen gas was fed to each of the pipes 11 - 17 at a flow rate of (L/6) sccm in pulse at a timing shown in Fig. 2. Into each pipe, the gas was fed for a time of T<sub>1</sub> and the gas was stopped for a time of T<sub>2</sub>. The partial pressure of DMAH was measured in the gas 7 flowing out from the outlet pipe 5 at varied T<sub>1</sub> and T<sub>2</sub>.

25 As shown in Table 1, even at the flow rate of hydrogen gas of 600 sccm, the DMAH was contained at a partial pressure of 1 Torr in the outgoing hydrogen gas, which is equal to the saturated vapor pressure at room temperature. The variation of flow rate of the outgoing hydrogen gas was within 1 %.

Table 1

L (sccm)	100	100	300	300	600	600	800	800
T <sub>1</sub> (sec)	30	60	30	60	30	60	30	60
T <sub>2</sub> (sec)	5	10	5	10	5	10	5	10
DMAH partial pressure (Torr)	1	1	1	1	1	1	0.8	0.8

#### Experimental example 2

Measurements were made in the same manner as in Experimental example 1 except that argon gas was used in place of hydrogen. The results were similar to those in Table 1. Even at the flow rate of 600 sccm, the DMAH was transported with the outgoing argon gas at a partial pressure equal to its saturated vapor pressure.

#### Experimental example 3

45 In Fig. 1, a metal pipe having five small openings was used as the tip of the respective pipes 11 - 17. Into an organometallic compound contained in the metallic container 10, hydrogen gas was introduced through a metallic pipe 4 at a flow rate of L sccm. By utilizing the gas switch 19, hydrogen gas was fed to each of the pipes 11 - 17 at a flow rate of (L/6) sccm in pulse at a timing shown in Fig. 2. Into each pipe, the gas was fed for time T<sub>1</sub> and the gas was stopped for time T<sub>2</sub>. The partial pressure of DMAH was measured in the gas 7 flowing out from the outlet pipe 5 at varied T<sub>1</sub> and T<sub>2</sub>.

50 As shown in Table 2, even at the flow rate of hydrogen gas of 3000 sccm, the DMAH was contained at a partial pressure of 1 Torr in the outgoing hydrogen gas, which is equal to the saturated vapor pressure at room temperature. The variation of flow rate of the outgoing hydrogen gas was within 1 %.

The carrier gas may be an inert gas, nitrogen gas, hydrogen gas, etc. For aluminum deposition, hydrogen gas is especially preferable since hydrogen atoms affect directly the aluminum deposition.

Table 2

L (sccm)	300	300	900	900	3000	3000	4000	4000
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Table 2 (continued)

T <sub>1</sub> (sec)	30	60	30	60	30	60	30	60
T <sub>2</sub> (sec)	5	10	5	10	5	10	5	10
DMAH partial pressure (Torr)	1	1	1	1	1	1	0.8	0.8

5

Experimental example 4

A gas-feeding system illustrated in Fig.3 was employed. In the device, the metal pipe 4 inserted in the organometallic compound had 100 small openings 5. Hydrogen gas 6 is introduced through the metal pipe 4 at a flow rate of L slm. The DMAH partial pressure contained in the hydrogen gas 7 flowing out from the outlet pipe 5 was measured.

As shown in Table 3, even at the flow rate of hydrogen gas of 10 slm, the DMAH was contained at a partial pressure of 1 Torr in the outgoing hydrogen gas, which is equal to the saturated vapor pressure at room temperature. The variation of flow rate of the outgoing hydrogen gas was within 1 %.

15

Table 3

L (slm)	0.5	1.5	5.0	10.0	12.0
DMAH partial pressure (Torr)	1	1	1	1	0.8

20

Experimental example 5

Measurements were made in the same manner as in Experimental example 4 except that argon gas was used in place of hydrogen. The results were similar to those in Table 3. Even at the flow rate of 10 slm, the DMAH was transported with the outgoing argon gas at a partial pressure equal to its saturated vapor pressure.

25

The gas-feeding system employed in this experimental example was found to be preferably applicable to a deposition-film-forming apparatus of vacuum CVD for selective deposition of an Al or Al-Si film of superior quality on a substrate as an electroconductive deposition film in the same way as the gas-feeding system employed in Experimental example 1.

30

Experimental example 6

A device illustrated in Fig. 4, in which five piezoelectric atomizers 34 were installed, was used for transporting an organometallic compound, DMAH. Hydrogen gas 6 is introduced through the metal pipe 37 at a flow rate of L slm. The DMAH partial pressure was measured of the hydrogen gas flowing out from the outlet pipe 9.

35

As shown in Table 4, even at the flow rate of hydrogen gas of 10 slm, the DMAH was contained at a partial pressure of 1 Torr in the flowing-out hydrogen gas, which is equal to the saturated vapor pressure at room temperature. The variation of flow rate of the outgoing hydrogen gas was within 1 %.

40

Table 4

L (slm)	0.5	1.5	5.0	10.0	12.0
DMAH partial pressure (Torr)	1	1	1	1	0.8

45

Experimental example 7

50

Measurements were made in the same manner as in Experimental example 6 except that argon gas was used in place of hydrogen. The results were similar to those in Table 4. Even at the flow rate of 10 slm, the DMAH was transported with the outgoing argon gas at a partial pressure equal to its saturated vapor pressure.

55

The gas-feeding device employed in this experimental example was found to be preferably applicable to a deposition-film-forming apparatus of vacuum CVD for selective deposition of an Al or Al-Si film of superior quality on a substrate as an electroconductive deposition film in the same way as the gas-feeding device employed in Experimental examples 1 - 5.

55 **Claims**

1. A system comprising a viscous organometallic compound and a gas-feeding device for feeding a starting gas comprising said compound for deposition-film-formation by a chemical vapour deposition method, said system

comprising:

a container (10) which contains the viscous organometallic compound (2) and has a space for forming an atmosphere of the starting gas comprising the compound;  
 5           gas-introducing means (4) connected to said container and having a plurality of gas-introducing openings (11-17) for introducing in use a carrier gas into said container to vaporise the compound, thereby producing the starting gas, said introducing means being arranged in use to introduce the carrier gas into the container at a flow rate of 100 sccm or less; and  
 10          accelerating-means (19) for accelerating the generation of the starting gas by controlling the introduction of the carrier gas so that in use the carrier gas is introduced into the plurality of gas-introducing openings (11-17) intermittently at a different timing for each opening.

2. The system of claim 1, wherein said accelerating means (19) comprises gas-switching means which in use turns on or turns off feed lines of the carrier gas for distributing the carrier gas through a turned-on feed line to its respective one of the plurality of gas-introducing openings.  
 15

3. A system comprising a viscous organometallic compound and a gas-feeding device for feeding a starting gas comprising said compound for deposition-film-formation by a chemical vapour deposition method, said system comprising:  
 20

a container (10) which contains the viscous organometallic compound (2) and has a space for forming an atmosphere of the starting gas comprising the compound;  
 gas-introducing means (4) connected to said container and having a plurality of gas-introducing openings (305) for introducing in use a carrier gas into said container to vaporise the compound, thereby producing the starting gas, said introducing means being arranged in use to introduce the carrier gas into the container at a flow rate of 100 sccm or less; and  
 25          accelerating-means (8) for accelerating the generation of the starting gas, said accelerating means being an ultrasonic oscillator which in use assists the formation of fine bubbles in the viscous organometallic compound (2).

30          4. A system comprising a viscous organometallic compound and a gas-feeding device for feeding a starting gas comprising said compound for deposition-film-formation by a chemical vapour deposition method, said device comprising:

35          a container (31) which contains the viscous organometallic compound;  
 a chamber (33) where in use an atmosphere of a starting gas comprising the compound is formed using the viscous organometallic compound from the container (31);  
 an inlet (37) into the chamber (33) for a carrier gas and an outlet (9) from the chamber (33) for the starting gas;  
 40          a plurality of nozzles (30) through which in use the viscous organometallic compound from the container (31) is fed into the chamber (33); and atomizing means (34) which converts the viscous compound being fed into the chamber (33) into a mist, the viscous compound flowing out with the carrier gas to provide said starting gas.

45          5. The system of claim 4, wherein piezoelectric atomisers (34) are provided for ejecting the organometallic compound through the nozzles (30).

6. The system device of any preceding claim, wherein the organometallic compound is dimethylaluminum hydride.

7. The system of any of claims 1-6 when used to produce a starting gas which contains dimethylaluminum hydride in a carrier gas which is hydrogen.  
 50

#### Patentansprüche

1. System mit einer viskosen organometallischen Verbindung und einer Gaszuführvorrichtung zum Zuführen eines die Verbindung enthaltenden Ausgangsgases zur Abscheidung eines Filmes durch ein chemisches Bedampfungsverfahren, wobei das System die folgenden Bestandteile umfaßt:  
 55

einen Behälter (10), der die viskose organometallische Verbindung (2) enthält und einen Raum zur Ausbildung

einer Atmosphäre des die Verbindung enthaltenden Ausgangsgases besitzt; eine Gaseinführreinrichtung (4), die mit dem Behälter verbunden ist und eine Vielzahl von Gaseinführöffnungen (11-17) aufweist, um im Betrieb ein Trägergas in den Behälter einzuführen und die Verbindung zu verdampfen, so daß auf diese Weise das Ausgangsgas erzeugt wird, wobei die Einführreinrichtung im Betrieb so angeordnet ist, daß sie das Trägergas in den Behälter mit einem Durchsatz von 100 sccm oder weniger einführt; und eine Beschleunigungseinrichtung (19) zum Beschleunigen der Erzeugung des Ausgangsgases durch Steuern der Einführung des Trägergases derart, daß im Betrieb das Trägergas intermittierend zu einem unterschiedlichen Zeitpunkt für jede Öffnung in die Vielzahl der Gaseinführöffnungen (11-17) eingeführt wird.

5 10 2. System nach Anspruch 1, bei dem die Beschleunigungseinrichtung (19) eine Gasumschalteinrichtung aufweist, die im Betrieb die Zuführleitungen des Trägergases öffnet oder absperrt, um das Trägergas durch eine geöffnete Zuführleitung auf die jeweilige Öffnung der Vielzahl der Gaseinführöffnungen zu verteilen.

15 15 3. System mit einer viskosen organometallischen Verbindung und einer Gaszuführvorrichtung zum Zuführen eines die Verbindung enthaltenden Ausgangsgases zur Abscheidung eines Filmes über ein chemisches Bedampfungsverfahren, wobei das System die folgenden Bestandteile umfaßt:

20 einen Behälter (10), der die viskose organometallische Verbindung (2) enthält und einen Raum zur Ausbildung einer Atmosphäre des die Verbindung enthaltenden Ausgangsgases besitzt;

25 eine Gaseinführreinrichtung (4), die mit dem Behälter verbunden ist und eine Vielzahl von Gaseinführöffnungen (305) aufweist, um im Betrieb ein Trägergas in den Behälter einzuführen und die Verbindung zu verdampfen, so daß auf diese Weise das Ausgangsgas erzeugt wird, wobei die Einführreinrichtung im Betrieb so angeordnet ist, daß das Trägergas in den Behälter mit einem Durchsatz von 100 sccm oder weniger eingeführt wird; und

25 eine Beschleunigungseinrichtung (8) zum Beschleunigen der Erzeugung des Ausgangsgases, bei der es sich um einen Ultraschall-Oszillator handelt, der im Betrieb zur Ausbildung von feinen Blasen in der viskosen organometallischen Verbindung (2) beiträgt.

30 4. System mit einer viskosen organometallischen Verbindung und einer Gaszuführvorrichtung zum Zuführen eines die Verbindung enthaltenden Ausgangsgases zur Abscheidung eines Filmes über ein chemisches Bedampfungsverfahren, wobei das System die folgenden Bestandteile umfaßt:

35 einen Behälter (31), der die viskose organometallische Verbindung enthält;

35 eine Kammer (33), in der im Betrieb eine Atmosphäre eines die Verbindung enthaltenden Ausgangsgases unter Verwendung der viskosen organometallischen Verbindung aus dem Behälter (31) erzeugt wird;

40 einen Einlaß (37) in die Kammer (33) für ein Trägergas und einen Auslaß (9) aus der Kammer (33) für das Ausgangsgas;

40 eine Vielzahl von Düsen (30) durch die im Betrieb die viskose organometallische Verbindung vom Behälter (31) in die Kammer (33) geführt wird; und

45 eine Zerstäubungseinrichtung (34), die die in die Kammer (33) geführte viskose Verbindung in einen Nebel umformt, wobei die viskose Verbindung mit dem Trägergas ausströmt, um das Ausgangsgas zur Verfügung zu stellen.

50 5. System nach Anspruch 4, bei dem piezoelektrische Zerstäuber (34) zum Ausstoßen der organometallischen Verbindung durch die Düsen (30) vorgesehen sind.

45 6. System nach einem der vorangehenden Ansprüche, bei dem die organometallische Verbindung Dimethylaluminiumhydrid ist.

50 7. Verwendung des Systems nach einem der Ansprüche 1 bis 6 zur Erzeugung eines Ausgangsgases, das Dimethylaluminiumhydrid in einem Trägergas enthält, das Wasserstoff ist.

#### Revendications

55 1. Système comprenant un composé organométallique visqueux et un dispositif d'alimentation en gaz destiné à fournir un gaz de lancement comprenant ledit composé pour la formation de films par dépôt par un procédé de dépôt chimique en phase vapeur, ledit système comprenant :

- un récipient (10) qui contient le composé organométallique visqueux (2) et dans lequel un espace est prévu pour la formation d'une atmosphère du gaz de lancement comprenant le composé ;  
 des moyens d'introduction de gaz (4) connectés audit récipient et ayant plusieurs ouvertures d'introduction de gaz (11-17) destinées à introduire en utilisation un gaz porteur dans ledit récipient pour vaporiser le composé, et générer ainsi le gaz de lancement, lesdits moyens d'introduction étant disposés en utilisation de façon à introduire le gaz porteur dans le récipient à un débit de 100 sccm ou moins, et  
 des moyens d'accélération (19) destinés à accélérer la génération du gaz de lancement en contrôlant l'introduction du gaz porteur de façon qu'en utilisation le gaz porteur soit introduit dans les diverses ouvertures d'introduction de gaz (11-17) de manière intermittente à un moment différent pour chaque ouverture.
2. Système selon la revendication 1, dans lequel lesdits moyens d'accélération (19) comprennent des moyens d'ouverture-fermeture d'arrivée de gaz qui, en utilisation, ouvrent ou ferment les conduites d'alimentation du gaz porteur pour distribuer le gaz porteur par une conduite d'alimentation ouverte sur son ouverture respective parmi la pluralité des ouvertures d'introduction de gaz.
3. Système comprenant un composé organométallique visqueux et un dispositif d'alimentation en gaz destiné à fournir un gaz de lancement comprenant ledit composé pour la formation de films par dépôt par un procédé de dépôt chimique en phase vapeur, ledit système comprenant :
- un récipient (10) qui contient le composé organométallique visqueux (2) et dans lequel un espace est prévu pour la formation d'une atmosphère du gaz de lancement comprenant le composé ;  
 des moyens d'introduction de gaz (4) connectés audit récipient et ayant plusieurs ouvertures d'introduction de gaz (305) destinées à introduire en utilisation un gaz porteur dans ledit récipient pour vaporiser le composé, et générer ainsi le gaz de lancement, lesdits moyens d'introduction étant disposés en utilisation de façon à introduire le gaz porteur dans le récipient à un débit de 100 sccm ou moins , et  
 des moyens d'accélération (8) destinés à accélérer la génération du gaz de lancement, lesdits moyens d'accélération étant un oscillateur ultrasonore qui, en utilisation, facilite la formation de bulles ténues dans le composé organométallique visqueux (2).
4. Système comprenant un composé organométallique visqueux et un dispositif d'alimentation en gaz destiné à fournir un gaz de lancement comprenant ledit composé pour la formation de films par dépôt par un procédé de dépôt chimique en phase vapeur, ledit système comprenant :
- un récipient (31) qui contient le composé organométallique visqueux;  
 une chambre (33) dans laquelle, en cours d'utilisation, une atmosphère d'un gaz de lancement comprenant le composé est formée en utilisant le composé organométallique visqueux contenu dans le récipient (31) ;  
 un orifice d'entrée (37) dans la chambre (33) pour un gaz porteur et un orifice de sortie (9) de la chambre (33) pour le gaz de lancement ;  
 plusieurs buses (30) à travers lesquelles, en cours d'utilisation, le composé organométallique visqueux contenu dans le récipient (31) est introduit dans la chambre (33) ; et des moyens d'atomisation (34) qui convertissent le composé visqueux introduit dans la chambre (33) en un embrun, le composé visqueux s'écoulant avec le gaz porteur pour fournir ledit gaz de lancement.
5. Système selon la revendication 4, dans lequel des atomiseurs piézo-électriques (34) sont fournis pour éjecter le composé organométallique à travers les buses (30).
6. Système selon l'une quelconque des revendications qui précèdent, dans lequel le composé organométallique est un hydrure de diméthylaluminium.
7. Système selon l'une quelconque des revendications 1 à 6 utilisé pour générer un gaz de lancement qui contient un hydrure de diméthylaluminium dans un gaz porteur qui est de l'hydrogène.

FIG. 1

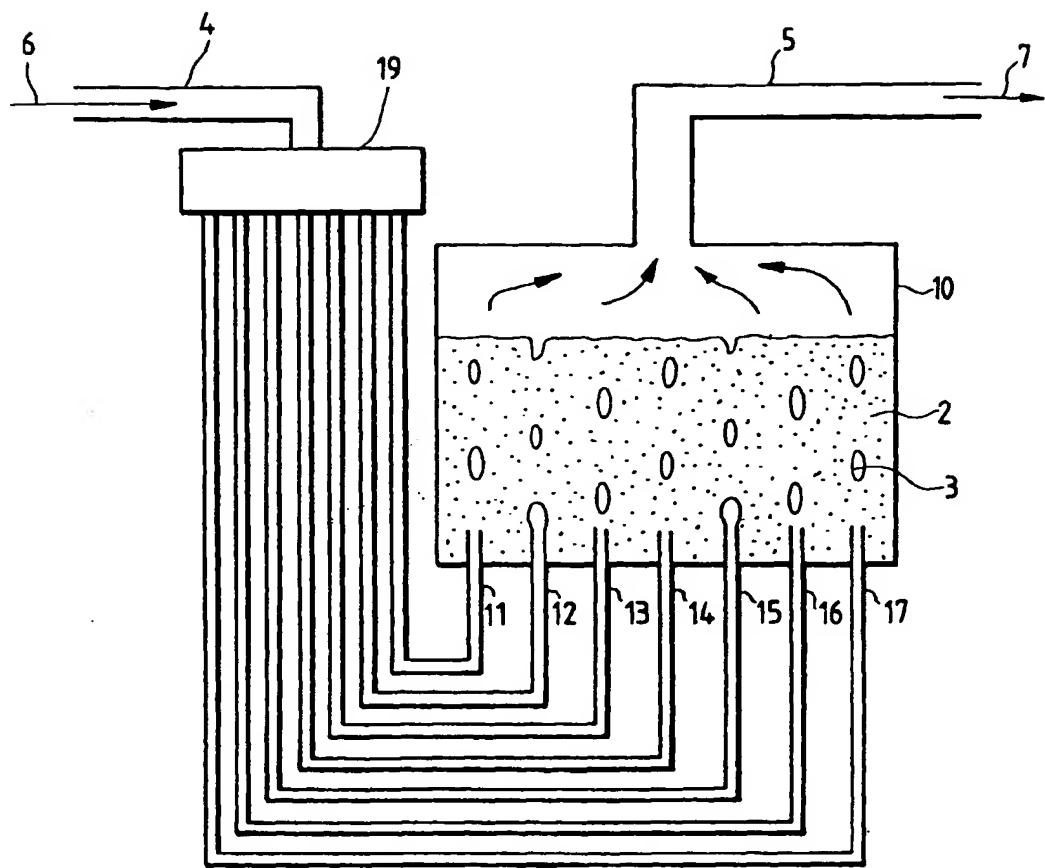


FIG. 2

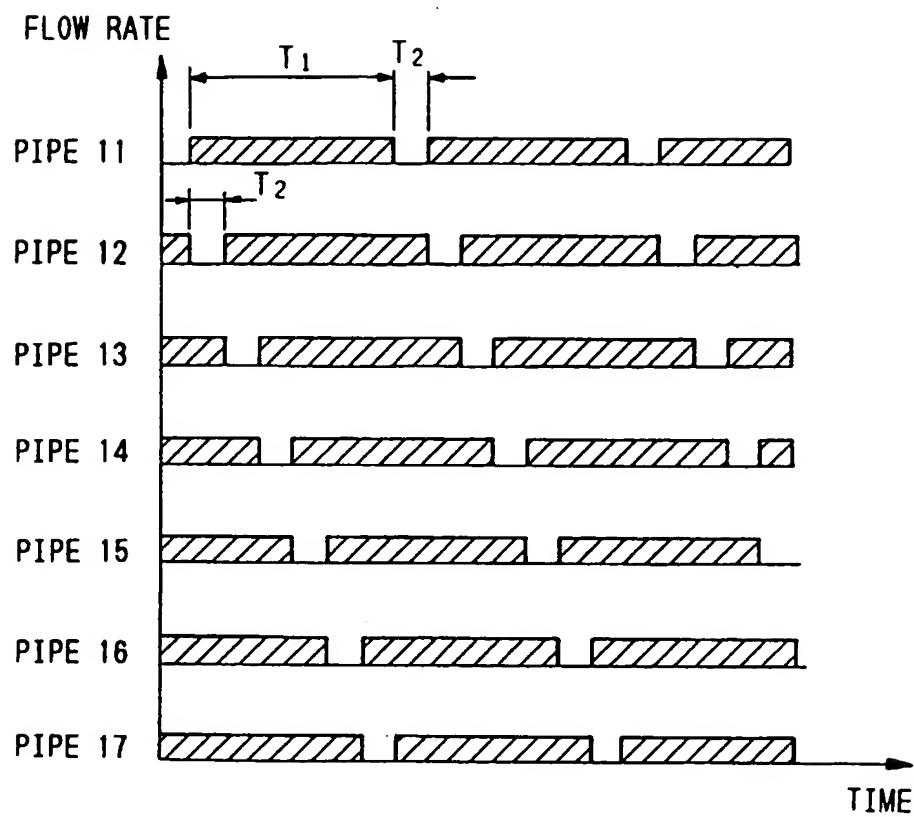


FIG. 3

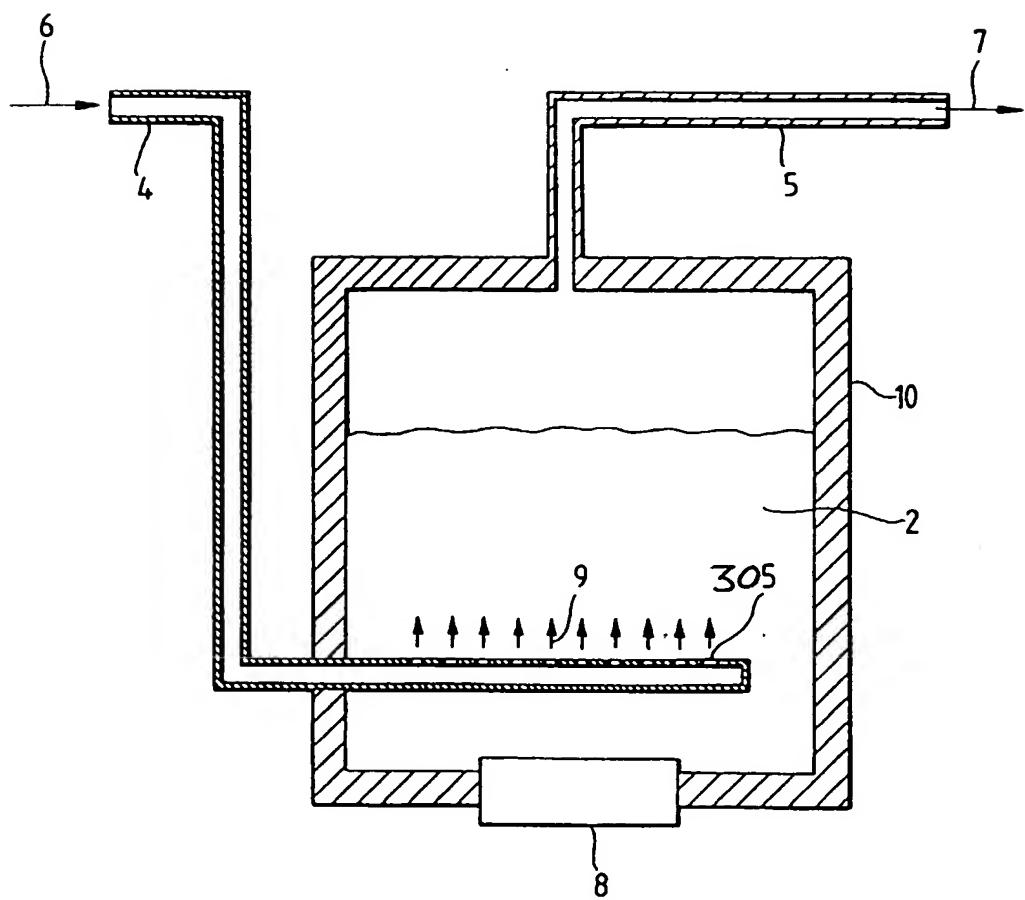


FIG. 4

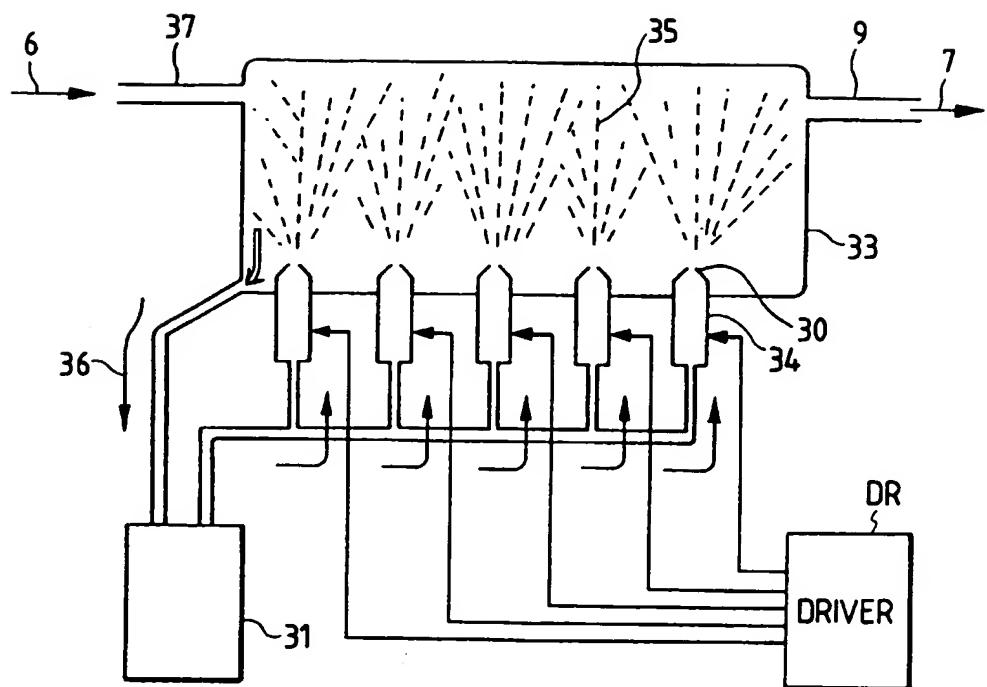


FIG. 5A

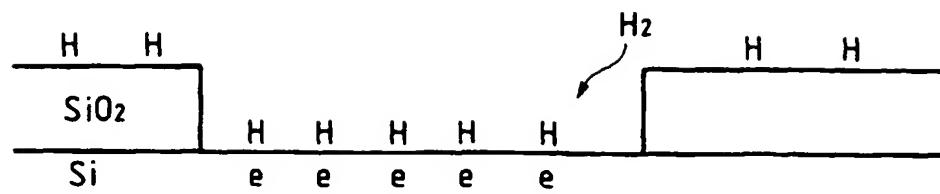


FIG. 5B

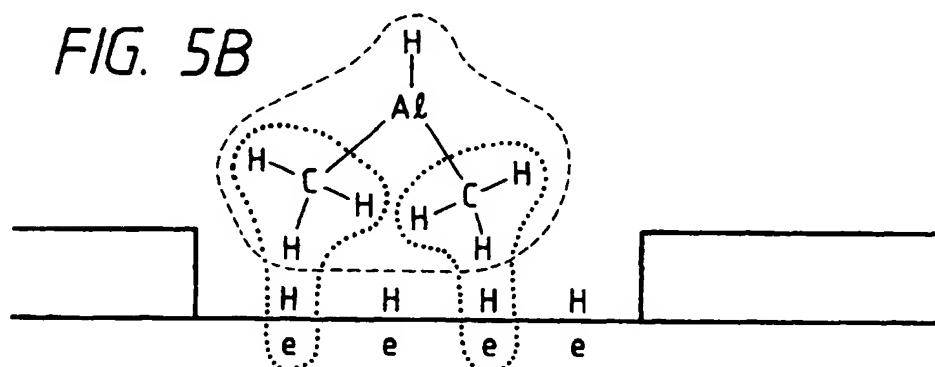


FIG. 5C

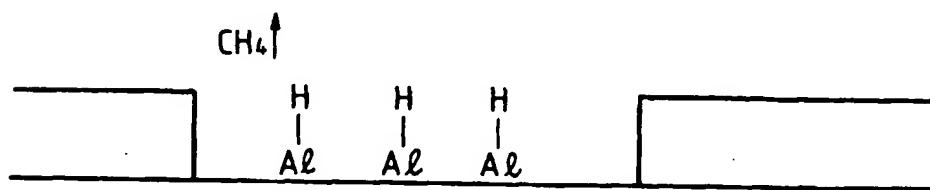


FIG. 5D

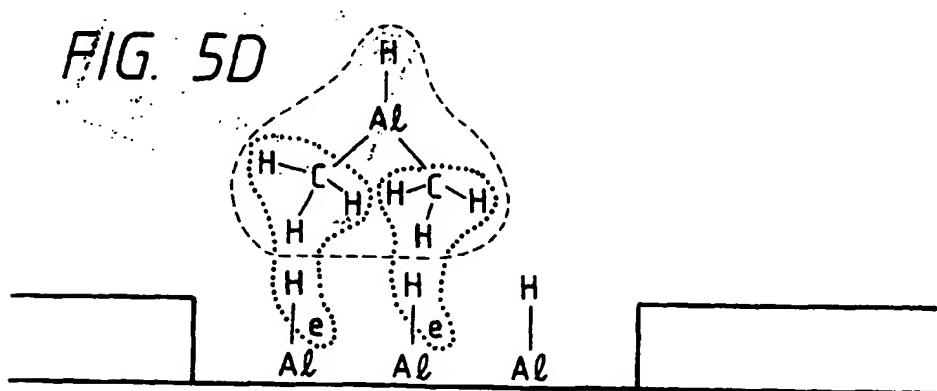


FIG. 6

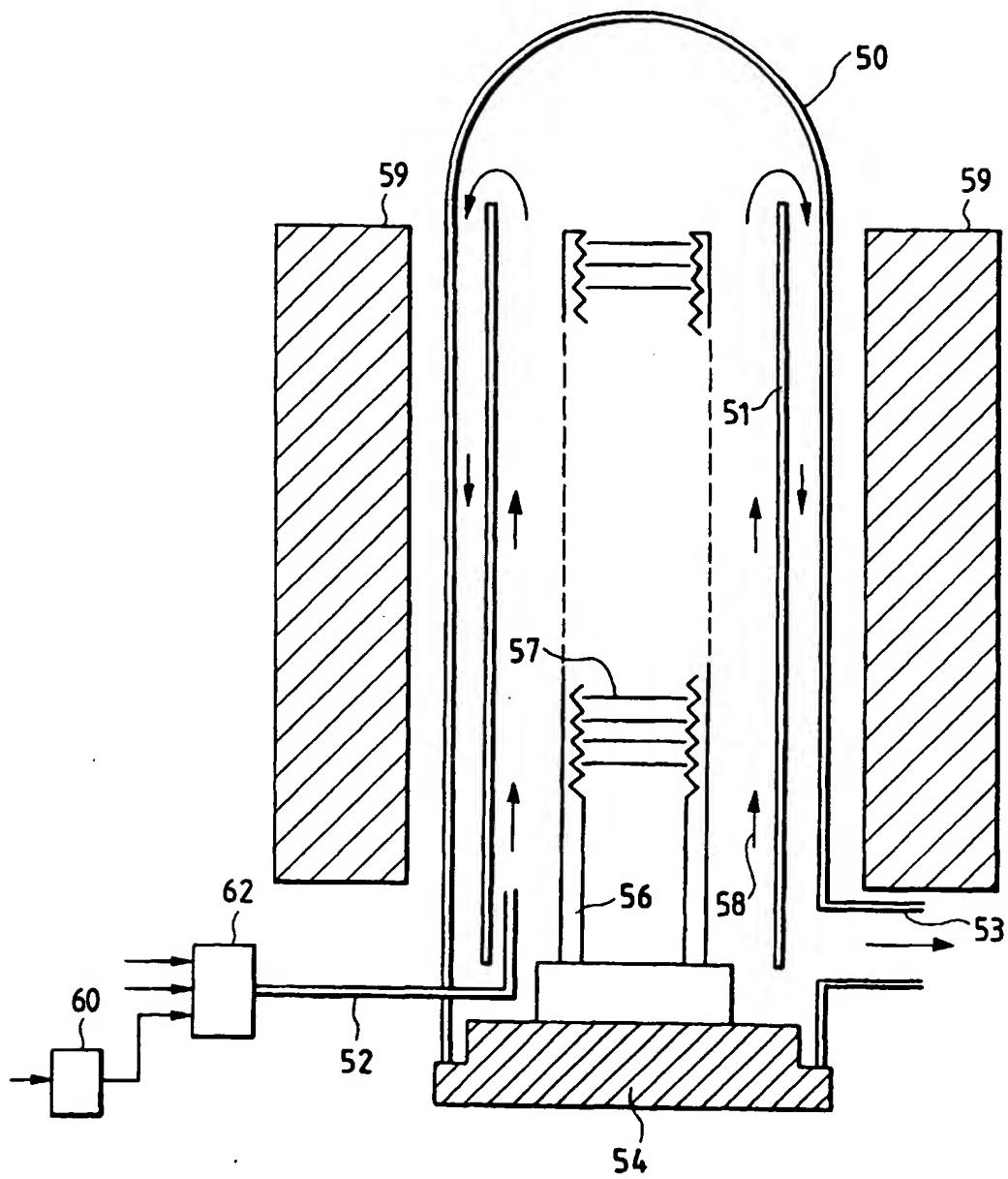


FIG. 7  
PRIOR ART

